7.—Identification of Western Australian columbite-tantalite, ixiolite and wodginite*

By M. W. Pryce†

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Abstract

In Western Australia, ordered columbite-tantalite occurs at Mt. Francisco, Nullagine, Greenbushes and Ravensthorpe, ixiolite at Tabba Tabba and Londonderry, wodginite at Wodgina, Tabba Tabba and Marble Bar and disordered columbite-tantalite at numerous localities.

The X-ray powder diffraction patterns of the mineral group are generally similar with diagnostic slight differences.

Ordered columbite-tantalite is orthorhombic, pale brown with vitreous lustre, good 010 cleavage and distinctive powder X-ray lines at 7.13Å and 5.30Å.

Partially to completely disordered columbite-tantalite is orthorhombic, black with submetallic lustre, no cleavage and the 7.13Å and 5.30Å lines are weak or absent but can be restored with heat treatment.

Ixiolite is orthorhombic, dark brown with resinous lustre, no cleavage and has no 7.13Å or 5.30Å line.

Wodginite is monoclinic, medium brown with resinous lustre, poor cleavage and distinctive X-ray powder pattern with numerous lines above 3.67Å and a strong doublet at 3.00Å and 2.97Å.

Introduction

In the past 15 years several hundred X-ray powder diffraction patterns have been made of various tantalum-niobium minerals at the W.A. Government Chemical Laboratories. Most of these minerals were in concentrates submitted for evaluation from the numerous widespread W.A. localities.

Prior to 1963 the majority of these patterns showing general similarity were classified under the general heading of "columbite-tantalite" following the classification of E. S. Simpson (1951, 1952). The variations in the patterns were not investigated as staff, equipment and time were not available.

One particularly unusual pattern with split major lines and described in the 1958 records as probably monoclinic was later shown to be of wodginite.

Wodginite was first described from Bernic Lake, Manitoba, Canada by Nickel, Rowland and McAdam in 1963 and named after the second locality Wodgina, W.A. (lat. 21°25′S. long. 118°30′E.)

The name was in recognition of the record by E. S. Simpson of "manganixiolite", which was actually wodginite from Wodgina in 1909.

The Canadian work also gave further credence to the new mineral olovotantalite, reinstated ixiolite, and introduced the terms ordered and disordered columbite-tantalite and pseudoixiolite. Disordered columbite-tantalite and pseudoixiolite are synonymous. The reader is referred to Nickel, Rowland and McAdam (1963 a & b) for powder x-ray data.

The X-ray patterns in the Laboratory file under "columbite-tantalite" have been reclassified into the suggested groups and examples found of each mineral except olovotantalite.

A well crystallised specimen from each group was selected for further work to apply the Canadian workers' techniques to W.A. specimens.

The study also correlated the colour, macrotransparency, lustre, crystallinity, cleavage and tin dioxide content of each mineral with the X-ray powder patterns.

A combination of the grain properties was found to be useful for initial recognition of the minerals in pegmatite or alluvium and a valuable supplement to X-ray and chemical data.

Mineral Properties, Occurrence and X-ray Crystallography

(a) *Ordered Columbite-Tantalite*

The X-ray powder pattern of ordered columbite-tantalite, (figure 1A) is distinguished from other "columbite-tantalite" patterns by the line of moderate intensity at 7.13Å (020) and the weak line at 5.30Å (110).

Two patterns, of specimen MDC 1266 from Nullagine (lat. 21°50′S. long. 120°7′E.) and specimen MDC 3233 from Ravensthorpe (lat. 33°35′S. long. 120°0′E.) showed both lines and were obviously of well crystallised mineral.

Two further occurrences were noted in 1967 in alluvial concentrates from Greenbushes (lat. 33°50′S. long. 116°0′E.) specimen MDC 550, and Mt Francisco. (lat. 21°20′S. long. 118°28′E.).

The crystals from specimens MDC 550, 1266 and 3233 are brown, elongated along the c axis and have a good 010 cleavage.

A rounded alluvial grain from Mt. Francisco was readily recognised as ordered columbite-tantalite by the 010 cleavage which even a high degree of roundness could not obscure.

Oscillation X-ray patterns of a small (0.1 mm) cleavage block from specimen MDC 1266 gave axial lengths approximating the published refined cell axes.

The cell is oriented in the crystals with the short cell dimension, c, along the crystal elongation and the long cell dimension, b, along the short crystal axis.

The oscillation X-ray pattern taken about the b axis showed a triple "super-cell" i.e. every third layer, h0l h3l h6l is equally strong and the intervening layers are all equally relatively

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† Government Chemical Laboratories, Perth, Western Australia.

weak. This repetition is the result of stacking three similar but not identical sub-cells along the b axis of the columbite-tantalite cell.

(b) *Ixiolite*

The X-ray powder pattern of ixiolite (figure 1E) can be distinguished from other "columbite-tantalite" patterns by the absence of any line with a d spacing greater than 3.65 Å (110).

Tin dioxide is considered to be an essential part of the composition of ixiolite.

Specimens MDC 89, MDC 273 and S389X from Londonderry (lat. 31°5′S. long. 121°7′E.) and MDC 2147 from Tabba Tabba (lat. 20°40′S. long. 118°52′E.) were chosen from the X-ray file because apart from the 6 Å line of microlite their patterns contained no lines above 3.65 Å.

Specimen MDC 2147, known to contain approximately 12 per cent tin dioxide was chosen for single crystal study. The sample is composed of numerous alluvial fragments and poorly shaped pseudo-tetragonal crystals slightly altered to microlite. The mineral is dark brown, brittle with a fine matte fracture surface and no cleavage. Macroscopically the grains are indistinguishable from cassiterite.

The crystal surfaces are usually strongly marked by the co-crystallised pegmatite minerals which have asserted their shape against the ixiolite. Mica flakes have imposed a pattern of deep diagonal grooves and pseudo cleavage "steps" on a number of crystals. Specimen MDC 273 and S389X were similar and had identical X-ray patterns. However, specimen MDC 89 was obviously not ixiolite as it was black, massive and contained no tin.

A large crystal 3 cm long and showing recognisable 010 and 001 forms with two rough faces possibly of the 110 prism was selected from specimen MDC 2147 for crystal study.

Oriented approximate cubes with 0.1 mm sides were formed by breaking up 0.1 mm thick sections cut perpendicular to the three crystal axis. The two ground surfaces of the cubes were a reference surface for approximate optical alignment on the goniometer head.

Calculations of cell dimensions from the oscillation X-ray patterns gave axial lengths approximating the refined cell dimensions given for ixiolite, Nickel, Rowland and McAdam (1963b).

(c) *Partially Disordered Columbite-Tantalite or Pseudoixiolite*

Most of the "columbite-tantalite" patterns in the Laboratory X-ray file have some trace of a line at 7.13 Å indicating partial disorder. The samples examined in detail have shown varying degrees of disorder in one crystal.

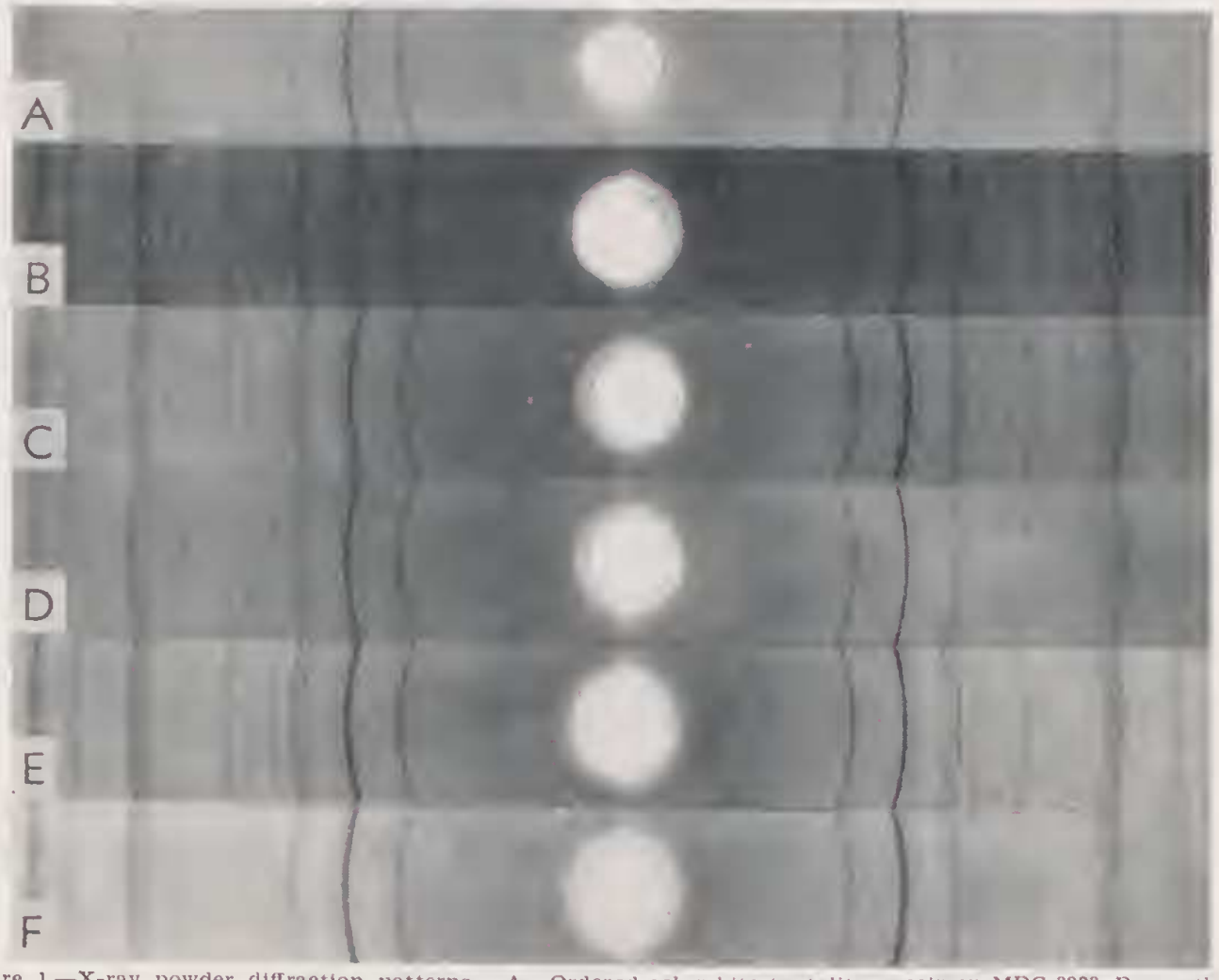

Figure 1.—X-ray powder diffraction patterns. A.—Ordered columbite-tantalite, specimen MDC 3233, Ravensthorpe. B.—Reordered columbite-tantalite (heated 1,000°C), specimen MDC 89, Londonderry. C.—Partially disordered columbite-tantalite, specimen MDC 618, Londonderry. D.—fully disordered columbite-tantalite, specimen MDC 89 Londonderry. E.—Ixiolite, specimen MDC 2147 Tabba Tabba. F.—Wodginite, specimen S388B Wodgina. Natural size. Camera diameter—114.83 mm Collimation—0.5 mm. Radiation—Cuk$_\alpha$.

TABLE 1

Comparative data of columbite-tantalite, ixiolite and wodginite from W.A.

Mineral	Colour	Macro transparency	Crystallinity	Lustre	Cleavage	Tin dioxide content approx. per cent	X-ray powder pattern
1. COLUMBITE-TANTALITE (a) Ordered	Pale brown	Translucent to transparent	Well shaped crystals often developed	Vitreous	good 010	low	Distinctive ; contains 7·13 Å and 5·30 Å lines
(b) Partially disordered	Black	Opaque	Well shaped crystals often developed, shape retained in alluvium	Sub-metallic	none	low	Similar to ordered pattern. Contains weak 7·13 Å line
(c) Fully disordered	Black	Opaque	Well shaped crystals often developed, shape retained in alluvium	Sub-metallic	none	low	Similar to ixiolite pattern. Re-orders on heating
2. IXIOLITE	Dark brown	Nearly opaque	Poorly shaped crystals. Shape often distorted by silicate minerals	Resinous	none	12	Distinctive : no line above 3·65 Å
3. WODGINITE	Medium brown	Translucent	Prismatic to acicular masses, well rounded in alluvium	Resinous	poor	varies up to 13	Distinctive : many lines above 3·67 Å, 3·00 Å and 2·97 Å doublet

Specimen MDC 618, (figure 1C) a large black crystal showing no cleavage was selected from the group. A chemical test proved it to contain only an insignificant amount of tin oxide. Thin sections were used to obtain oriented cubes for the oscillation camera.

The rotation pattern about the b axis showed faint remnants of the layers h1l, h2l, h4l, and h5l, verifying the partial disorder along this axis.

Specimen MDC 89, (figure 1D.) previously rejected from the ixiolite group, was also studied by X-ray oscillation patterns but showed no trace of h1l, h2l, h4l, and h5l indicating that the sample is completely disordered.

On heating for 1 hour at 1000°C specimen MDC 89 changed colour from black to brown and the X-ray pattern (figure 1B.) showed that it had re-ordered.

(d) *Wodginite*

The powder pattern of wodginite (figure 1 F) can be readily recognised by the numerous lines above 3.67 Å.

Wodginite has been recorded from three W.A. localities:—

(i) Wodgina, specimens S388B and MDC 2782-3 in pegmatite.

(ii) Tabba Tabba, specimens S3315 and specimen MDC 2259-60 from eluvial concentrates.

(iii) Marble Bar (lat. 21°5′S. long. 119°40′E) Lab. No. 9846/58 from alluvium.

A further specimen (Lab. No. 8800/66), was submitted for evaluation in 1966 from an unspecified locality, probably in the Wodgina area. The sample was representative of a commercial parcel of wodginite and consisted of brown well-rounded grains with the appearance of alluvial monazite. The alluvial wodginite in specimen Lab. No. 9846/58 from Marble Bar is identical with specimen Lab No. 8800/66 in appearance.

The wodginite in specimens S388B and MDC 2782-3 from Wodgina consists of brown resinous crystals with prismatic to acicular habit in pegmatite. The mineral is distinguished by poor cleavage from ordered columbite-tantalite, by colour from disordered columbite-tantalite and by habit from ixiolite and cassiterite.

The mineral from Tabba Tabba, specimen S3315 and MDC 2259-60 consists of eluvial pebbles of pale brown wodginite intergrown with and veined by white simpsonite. The pebbles are thickly coated with microlite which in some pieces has pseudomorphed the hexagonal shape of the replaced simpsonite.

Specimen S3315 was collected from Tabba Tabba in 1929 possibly at M.L.312, site of the first simpsonite occurrence.

Single crystal studies of wodginite were not attempted since Nickel et al (1963a) have presented full data on material identical with the Wodgina specimens S388B and MDC 2782-3. Positive identification of wodginite is still dependent on its distinctive X-ray pattern.

Discussion

To date only a broad initial survey with intensive work on a few specimens has been done with W.A. material. The findings of Nickel, Rowland and McAdam (1963a & b) appear to apply to the W.A. minerals examined. Useful properties for initial sorting and approximate identification of the minerals before X-raying have been noted and statements about their frequency of occurrence can be made.

Nearly all the W.A. columbite-tantalite is partially disordered. The powder pattern is recognised by the weakness of the 7.13 Å line and the absence of the 5.30 Å line. Typical specimens are black, opaque, with a submetallic lustre and no cleavage. Most material exhibits some crystal shape, particularly the 010 pinacoid faces so that some samples contain many thin rectangular plates.

Specimen MDC 89 has been recorded as a completely disordered columbite-tantalite with an X-ray pattern nearly identical with ixiolite.

Ordered columbite-tantalite has been recorded from only four W.A. localities. The X-ray pattern is distinguished by the two lines at 7.13 Å and 5.30 Å. The mineral is pale brown, has a high transparency, and a good 010 cleavage.

Ixiolite has been recorded from only two W.A. localities. The ixiolite X-ray pattern is distinguished by having no lines above 3.65 Å. The mineral is dark brown, with no cleavage, usually shows deep markings and contains tin dioxide as an essential constituent.

Wodginite has been recorded from only three localities in W.A.

The wodginite X-ray pattern is recognised by the numerous lines above 3.67Å and the splitting of the major line at 3.00 Å. It is brown with poor cleavage, shows no crystal shape in alluvium and may contain up to 13 per cent of tin dioxide.

The accompanying Table 1 sets out the distinguishing features of the minerals.

Prints of X-ray patterns of typical specimens, figure 1, are also included.

The patterns were taken with a 114.83 mm diameter camera using CuK_α radiation and 0.5 mm collimation.

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